NOV 2 6 1968

NATIONAL BUREAU OF STANDARDS REPORT

9946

STRESS-CORROSION BEHAVIOR OF TI-6AL-4V ALLOY IN A SYNTHETIC SEA WATER ENVIRONMENT

To

Naval Ship Research and Development Center
Department of the Navy
Carderock, Maryland

NBS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards[1] was established by an act of Congress March 3, 1901. Today, in addition to serving as the Nation's central measurement laboratory, the Bureau is a principal focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. To this end the Bureau conducts research and provides central national services in three broad program areas and provides central national services in a fourth. These are: (1) basic measurements and standards, (2) materials measurements and standards, (3) technological measurements and standards, and (4) transfer of technology.

The Bureau comprises the Institute for Basic Standards, the Institute for Materials Research, the Institute for Applied Technology, and the Center for Radiation Research.

THE INSTITUTE FOR BASIC STANDARDS provides the central basis within the United States of a complete and consistent system of physical measurement, coordinates that system with the measurement systems of other nations, and furnishes essential services leading to accurate and uniform physical measurements throughout the Nation's scientific community, industry, and commerce. The Institute consists of an Office of Standard Reference Data and a group of divisions organized by the following areas of science and engineering:

> Applied Mathematics—Electricity—Metrology—Mechanics—Heat—Atomic Physics—Cryogenics[2]—Radio Physics[2]—Radio Engineering[2]—Astrophysics[2]—Time and Frequency.[2]

THE INSTITUTE FOR MATERIALS RESEARCH conducts materials research leading to methods, standards of measurement, and data needed by industry, commerce, educational institutions, and government. The Institute also provides advisory and research services to other government agencies. The Institute consists of an Office of Standard Reference Materials and a group of divisions organized by the following areas of materials research:

> Analytical Chemistry—Polymers—Metallurgy — Inorganic Materials — Physical Chemistry.

THE INSTITUTE FOR APPLIED TECHNOLOGY provides for the creation of appropriate opportunities for the use and application of technology within the Federal Government and within the civilian sector of American industry. The primary functions of the Institute may be broadly classified as programs relating to technological measurements and standards and techniques for the transfer of technology. The Institute consists of a Clearinghouse for Scientific and Technical Information,[3] a Center for Computer Sciences and Technology, and a group of technical divisions and offices organized by the following fields of technology:

> Building Research—Electronic Instrumentation — Technical Analysis — Product Evaluation—Invention and Innovation— Weights and Measures — Engineering Standards—Vehicle Systems Research.

THE CENTER FOR RADIATION RESEARCH engages in research, measurement, and application of radiation to the solution of Bureau mission problems and the problems of other agencies and institutions. The Center for Radiation Research consists of the following divisions:

> Reactor Radiation—Linac Radiation—Applied Radiation—Nuclear Radiation.

---

[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D. C. 20234.
[2] Located at Boulder, Colorado 80302.
[3] Located at 5285 Port Royal Road, Springfield, Virginia 22151.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

3120647

NOV 26 1968

NBS REPORT

9946

STRESS-CORROSION BEHAVIOR OF TI-6AL-4V ALLOY IN A SYNTHETIC SEA WATER ENVIRONMENT

By

W. F. Gerhold
and
B. Z. Weiss

To

Naval Ship Research and Development Center
Department of the Navy
Carderock, Maryland

IMPORTANT NOTICE

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Stress-Corrosion Behavior of Ti-6Al-4V Alloy in a Synthetic Sea Water Environment

Reference: (a) Department of the Navy, Naval Ship Research and Development Center, Carderock, Maryland.

Introduction: It has been known for several years that, given the proper set of conditions, some titanium alloys are susceptible to stress-corrosion cracking. Stress-corrosion cracking may occur in sea water if there is a flaw of sufficient size and acuity and if the stress reaches a certain critical value. The mechanism of environmental cracking is not completely understood, but it has been postulated that the following may play a decisive role.

1. Corrosion by chemical attack by one or more chemical species at particular structural sites that are sensitive to stress, such as at stacking faults, grain boundaries or dislocation pile-ups.

2. Embrittlement by hydrogen whereby in the presence of free hydrogen there are formed brittle phase products or where there is an impedance of plastic deformation due to the pinning of dislocations by hydrogen in solution in the material.

3. The adsorption of certain ions along particular crystallographic planes which results in a reduction of the surface energy due to a weakening effect on the cohesive bond at these areas.

4. Rupture of the thin protective surface film on the metal which may trigger any or all three of the previous mechanisms.

Material: Eighty-eight (88) specimens that had been machined from 1/2 in. and 2 in. thick Ti-6Al-4V alloy plate were submitted by Reference (a). The specimens included samples from base-metal areas, weld areas and areas from welded material in the heat affected zone.

Specimens machined from the 2 in. thick plate had been obtained from areas on the plate at the surface and at areas in the center of the thickness of the plate.

Some specimens of the base-metal material from the 2 in. thick plate were oriented with their long axis in a direction longitudinal to the direction of rolling and others with their long axis transverse to the direction of rolling. Information relative to the orientation of specimens machined from the 1/2 in. thick plate was not available.

Details of the distribution and identification of specimens machined from plate of either thickness are given in Table 1.

Specimen Preparation: As noted above, all specimens in the form of rectangular bars approximately 3/4 in. wide x 7/16 in. thick had been machined to the test configuration prior to receipt by NBS. A sharp "V" notch (stress raiser) had been machined at the mid-length across one face of each specimen. Side grooves in the form of "V" notches had been machined across the two adjacent sides of the specimen. These latter notches were provided to reduce the size of shear lips that may form during test at the time of fracture. In order to increase the acuity of the stress raiser, a fatigue crack was introduced at the base of the "V" notch.

The apparatus used in this investigation to fatigue crack the specimens is shown in Figures 1 and 2. The crank and lever system from a Krause fatigue testing machine was used to apply a bending load on the specimen, with the specimen acting as a cantilever beam. The specimen was clamped, notched face down, rigidly at one end. A ball shaped anvil located toward the other end of the specimen was fastened to the lever which in turn was connected to the crank. A cyclic bending stress was applied to the specimen at the notched area by the anvil acting through the crank and lever system. Repeated cycling resulted in the initiation of a fatigue crack at the notch. The depth of the fatigue crack on both sides of the specimen was measured with the aid of a microscope.

Procedure: The test method used in this investigation was similar to that introduced by B. F. Brown.(1) In this method a precracked specimen is stressed in bending as a cantilever beam. Figure 3 shows the test apparatus used. The specimen is enclosed at the notched area in a plastic container (stress-corrosion cell). It is then clamped at one end, notched face up, to a rigid post. The other end is clamped to a long beam at the end of which is a container to which the load is applied. The corrodent (synthetic sea-water solution ASTM D-1141-52) is pumped from a reservoir through plastic tubing into the stress-corrosion cell. A return system is provided, at the bottom of the cell, through which the solution is returned to the reservoir. When the solution has reached a level sufficient to cover the specimen (this is controlled by a pet cock in the return line), the load is applied by adding weights to the container at the end of the cantilever beam.

The applied stress which is dependent upon the size of the flaw (stress raiser) is expressed in terms of the fracture mechanics stress intensity factor, K_I. For a rectangular precracked notched bar, this factor can be calculated from the equation from Kies, et al.(2)

$$K = \frac{4.12\, M \sqrt{\frac{1}{\alpha^3} - \alpha^3}}{BD^{3/2}}$$

where: $\alpha = 1 - \frac{a}{D}$

a = Notch depth + depth of fatigue crack.
D = Vertical depth of the specimen.
B = Horizontal depth of the specimen.
M = Bending moment at the notch.

If measurements are in inches and if the moment is expressed in in.-lbs, the stress intensity factor has the units Ksi $\sqrt{\text{in.}}$

For purposes of this investigation two quantities of stress intensity were determined. One designated K_{I_x} (the stress intensity required for fracture in air) is a rough approximation of the toughness of the material. The second designated $K_{I_{scc}}$ is the threshold stress intensity factor for a specific corrosive environment, below which stress-corrosion cracking will not occur.

The pertinent dimensions of all specimens tested in this investigation were determined with a micrometer caliper. The depth of the "V" notch was measured with the aid of a toolmaker's microscope and the depth of the fatigue crack was determined with the aid of the microscope previously mentioned.

One specimen from each group of 4 was used for determining the K_{I_x}. A precracked specimen was enclosed at the notched area by a plastic container and placed in the stress apparatus. Anhydrous $CaSO_4$ crystals were added to the container and the container was then sealed. The $CaSO_4$ was used to obtain a relatively dry-air condition. A load was then applied at the end of the beam. This load was increased until fracture of the specimen occurred. After fracture, calculations were made, using the above equation, to obtain the stress intensity factor required for fracture in air, K_{I_x}. The quantity determined for each group of specimens is given in Table 2.

Two specimens from each group of 4 were used to determine the approximate $K_{I_{scc}}$. Each one of these specimens was precracked, enclosed by the stress-corrosion cell and then stressed in the sea salt enviroment at a known stress intensity level, K_I . If failure did not occur within one hour, the specimen was removed from test and returned to the fatigue testing machine. The depth of the fatigue crack was increased slightly in order to provide a fresh sharp notch at the base of the crack. (Other investigators have reported that possibly due to creep effects, some blunting of the crack occurs during the stress corrosion tests.) The newly precracked specimen was then returned to

the stress apparatus and stressed again at a slightly higher stress intensity. This was repeated until failure occurred or until the depth of the fatigue crack had increased to a value which made the specimen unsuitable for further tests. Figure 4 shows the uniformity of the fatigue cracks initiated by the precracking process and typical fractured surfaces for 3 failed stress-corrosion test specimens.

Generally, a stress intensity factor of 50 Ksi $\sqrt{in.}$ was used as the initial level and this was increased in 10 Ksi $\sqrt{in.}$ steps until the specimen either fractured or was removed from test. By comparing the stress intensity factor for both specimens at which fracture occurred an approximate $K_{I_{scc}}$ was determined. If fracture of only one specimen was obtained, then the stress intensity factor at which fracture occurred was assumed to be the approximate $K_{I_{scc}}$.

After the approximate $K_{I_{scc}}$ was determined for each group of specimens, a fourth specimen was used to determine the apparent $K_{I_{scc}}$ A maximum of 3 tests were performed on each specimen by alternately precracking and stressing the specimen as noted above. The intensity level chosen for the first test on the fourth specimen was at least 10 Ksi $\sqrt{in.}$ below the approximate $K_{I_{scc}}$. This was increased in 5 Ksi $\sqrt{in.}$ steps until the stress intensity level during test was 5 Ksi $\sqrt{in.}$ above the approximate $K_{I_{scc}}$.

Results: The results obtained from an investigation of the stress-corrosion behavior of Ti-6Al-4V alloy in a synthetic sea-water environment are summarized in Table 2. Data are given for individual specimens in terms of the maximum stress intensity, K_I at which no failure occurred as well as the maximum K_I and time at which the specimen did fail. In interpreting the data obtained to establish a threshold stress intensity $\left(K_{I_{scc}}\right)$ below which failure will not occur, it is necessary to take into account both the K_I and time to produce failure. The data, based solely upon K_I at failure, could lead to the establishment of an erroneous $K_{I_{scc}}$ value. For this reason in some instances there are two different values given for the $K_{I_{scc}}$, the apparent and the probable. The latter is considered to be more reliable.

The $K_{I_{scc}}$ for specimens obtained from base metal areas and heat affected areas of weldments was found to be between 75 and 80 Ksi $\sqrt{in.}$ There was no apparent difference for plate of either thickness. The stress relieving heat treatment had no noticeable effect on the $K_{I_{scc}}$ value obtained for specimens from either one of these areas. Specimen orientation had no effect upon the $K_{I_{scc}}$ of the base metal.

The stress relieving heat treatment did substantially decrease the $K_{I_{scc}}$ (60 Ksi $\sqrt{in.}$) at the weld area of the specimens machined from surface areas of the 2 in. thick plate. At similar areas on specimens that had not been stress relieved there was a slight increase in the $K_{I_{scc}}$ (85 Ksi $\sqrt{in.}$). There was also a slight decrease in the $K_{I_{scc}}$ (70 Ksi $\sqrt{in.}$) for specimens machined from weld areas of the 1/2 in. thick plate.

Conclusions: It has been demonstrated that immunity to stress-corrosion can be defined in terms of a threshold stress intensity, $K_{I_{scc}}$. The threshold stress intensity is that quantity, expressed in units of Ksi $\sqrt{in.}$, below which a material will not fail by stress-corrosion cracking, in a given corrosive medium.

Comparison of the $K_{I_{scc}}$ values obtained from stress-corrosion tests conducted in this investigation revealed the following.

1. Base-metal material and material obtained from weldments in the heat affected zone.

    a. The $K_{I_{scc}}$ was between 75 and 80 Ksi $\sqrt{in.}$

    b. Thickness of the plate (1/2-in. vs 2-in.) had no effect on the $K_{I_{scc}}$.

    c. Similarly a stress-relieving heat treatment had no effect on the $K_{I_{scc}}$.

2. Base-metal material.

    a. Orientation (longitudinal vs transverse) of specimens obtained from 2 in. thick plate had no effect in either increasing or decreasing the $K_{I_{scc}}$.

3. Weld-metal material.

    a. The $K_{I_{scc}}$ at weld-metal areas of stress-relieved material was lower. This decrease was substantially greater for specimens machined from the surface areas of the 2-in. thick plate (60 Ksi $\sqrt{in.}$) than for specimens machined from the 1/2-in. thick plate (70 Ksi $\sqrt{in.}$).

    b. There was an increase in the $K_{I_{scc}}$ (85 Ksi $\sqrt{in.}$) for specimens machined from the surface areas of non-stress relieved 2-in. thick plate.

REFERENCES

(1) B. F. Brown, "A New Stress-Corrosion Cracking Test for High Strength Alloys," Materials Research and Standards, Vol. 6, No. 3 (1966).

(2) J. A. Kies, et al., "Fracture Testing of Weldments," In, "Fracture Toughness Testing and It's Applications," ASTM Special Technical Publication 381, 1965, pp. 328-356.

Table 1. Distribution and identification of specimens machined from 1/2 in. and 2 in. thick plate.

Coupon number	Heat treatment (a)	Specimen orientation (b)	Plate thickness, in.	Number of specimens		
				Base metal	Weldments	
					Weld area	Heat affected zone
S	SR	-	1/2	-	4	4
K	SR	-	1/2	4	-	-
L	None	-	1/2	-	4	4
V	None	-	1/2	4	-	-
A	SR	L, surface	2	4	4	4
A	SR	L, surface	2	4	4	4
A	SR	T, surface	2	4	-	-
A	SR	T, center	2	4	-	-
B	None	L, surface	2	4	4	4
B	None	L, center	2	4	4	4
B	None	T, surface	2	4	-	-
B	None	T, center	2	4	-	-

(a) Heat treatment after fabrication -- SR = stress relieved. Prior heat treatment, not reported.

(b) L - longitudinal, T - transverse, surface - machined from surface area of plate and center - machined from areas at center of thickness of plate.

Table 2. Results obtained from stress-corrosion tests conducted on Ti-6Al-4V alloy plate specimens.

Coupon number	Plate thickness, in.	Heat treatment (a)	Specimen type (b)	Test area (c)	Direction of specimen (d)	K_{Ix} (in air), Ksi $\sqrt{in.}$	K_I (sea water),max, Ksi $\sqrt{in.}$ (g)		Time to failure, min	$K_{I_{scc}}$ - Ksi $\sqrt{in.}$	
							No failure (e)	Failure		Probable	Apparent
A	2	SR	BM	Surface	L	(f)				80	80
							45.6	-	-		
							124.8	-	-		
							78.0	-	-		
							82.1	85.8	21		
A	2	SR	BM	Center	L	100.6				75	80
							80.3	88.6	0		
							-	85.2	45		
							80.2	81.8	1		
A	2	SR	HAZ	Surface	L	96.2				80	85
							80.7	89.7	17		
							84.9	-	-		
							84.7	-	-		
A	2	SR	HAZ	Center	L	85.9				75	75
							59.7	110.0	0		
							-	89.0	0		
							74.9	79.1	1		
A	2	SR	W	Surface	L	96.5				60	60
							69.9	84.3	0		
							65.2	65.0	0		
							64.3	-	-		
A	2	SR	W	Center	L	89.2				75	75
							70.3	79.1	0		
							75.3	-	-		
							74.7	-	-		

Table 2. Continued

Coupon number	Plate thickness, in.	Heat treatment (a)	Specimen type (b)	Test area (c)	Direction of specimen (d)	K_{Ix} (in air), Ksi $\sqrt{in.}$	K_I (sea water),max, Ksi $\sqrt{in.}$ (g)		Time to failure, min	$K_{I_{scc}}$ - Ksi $\sqrt{in.}$	
							No failure (e)	Failure		Probable	Apparent
A	2	SR	BM	Surface	T	100.5	90.2 - 79.6	90.1 84.9 82.5	3 1 0	75	80
A	2	SR	BM	Center	T	105.2	79.3 - 74.9	89.6 81.9 80.0	53 0 58	75	80
B	2	None	BM	Surface	L	94.8	99.4 74.6 80.2	101.8 80.1 -	0 44 -	75	80
B	2	None	BM	Center	L	100.0	90.6 90.3 79.8	99.0 92.4 84.8	2 0 2	80	80
B	2	None	HAZ	Surface	L	93.8	80.5 - 84.9	89.4 85.1 -	1 12 -	80	80
B	2	None	HAZ	Center	L	98.8	110.1 79.7 84.7	118.1 85.5 -	0.5 34 -	80	80

Table 2. Continued

Coupon number	Plate thickness, in.	Heat treatment (a)	Specimen type (b)	Test area (c)	Direction of specimen (d)	K_{Ix} (in air), Ksi $\sqrt{in.}$	K_I (sea water), max, Ksi $\sqrt{in.}$ (g)		Time to failure, min	$K_{I_{scc}}$ - Ksi $\sqrt{in.}$	
							No failure (e)	Failure		Probable	Apparent
B	2	None	W	Surface	L	96.0	80.4 85.2 65.9	89.4 90.1 -	1 1 -	85	85
B	2	None	W	Center	L	91.8	70.6 84.9 66.8	- 89.5 87.3	- 0.5 0	80	80
B	2	None	BM	Surface	T	92.5	80.7 - 77.2	90.5 84.6 86.2	0 1 16	80	80
B	2	None	BM	Center	T	92.1	89.0 79.8 75.1	99.5 84.9 79.7	0 39 2	75	75
S	1/2	SR	W	-	-	88.2	69.4 80.1 -	79.7 84.8 74.7	0 1 1	70	70
S	1/2	SR	HAZ	-	-	105.9	79.8 84.7 79.9	89.3 90.0 84.7	2 11 55	80	80
K	1/2	SR	BM	-	-	104.2	98.5 84.5 79.6	109.1 90.8 84.6	0 0.5 1	80	80

Table 2. Continued

Coupon number	Plate thickness, in.	Heat treatment (a)	Specimen type (b)	Test area (c)	Direction of specimen (d)	K_{Ix} (in air), Ksi $\sqrt{in.}$	K_I (sea water),max,[g] Ksi $\sqrt{in.}$		Time to failure, min	$K_{I_{scc}}$ - Ksi $\sqrt{in.}$	
							No failure (e)	Failure		Probable	Apparent
L	1/2	None	W	-	-	100.3	80.4	90.1	1	75	75
							80.3	85.6	2		
							80.3	77.0	0		
L	1/2	None	HAZ	-	-	91.4	80.6	89.8	0.5	75	75
							79.9	84.6	1		
							74.9	78.1	0		
V	1/2	None	BM	-	-	92.4	79.7	90.1	1	80	80
							79.8	84.8	1		
							84.7	-	-		

(a) SR - stress relieved. None - no additional heat treatment. Prior heat treatment unknown.
(b) BM - base metal. HAZ - heat affected zone. W - Weld area.
(c) Specimens machined from plate at areas shown. Center refers to the area at the middle of the plate thickness.
(d) Specimens machined from plate such that the long axis with respect to the direction of rolling was longitudinal (L) or transverse (T).
(e) Duration of test was 60 minutes (max).
(f) Not determined.
(g) Synthetic sea water solution (ASTM D-1141-52).

Figure 1. Modified Krause fatigue testing machine used to precrack the notched Ti-6Al-4V alloy plate specimens. Specimen, arrow E is clamped rigidly at A. Eccentric motion of crank, arrow F, actuates lever C which in turn moves the anvil B. Action of the anvil is in a vertical direction (up and down) against the specimen at arrow E resulting in an alternating bending stress being applied at the notched area of the specimen. Repeated cycling action of the anvil against the specimen induces a fatigue crack at the notch. Microscope, arrow D, is used to measure the depth of the crack.

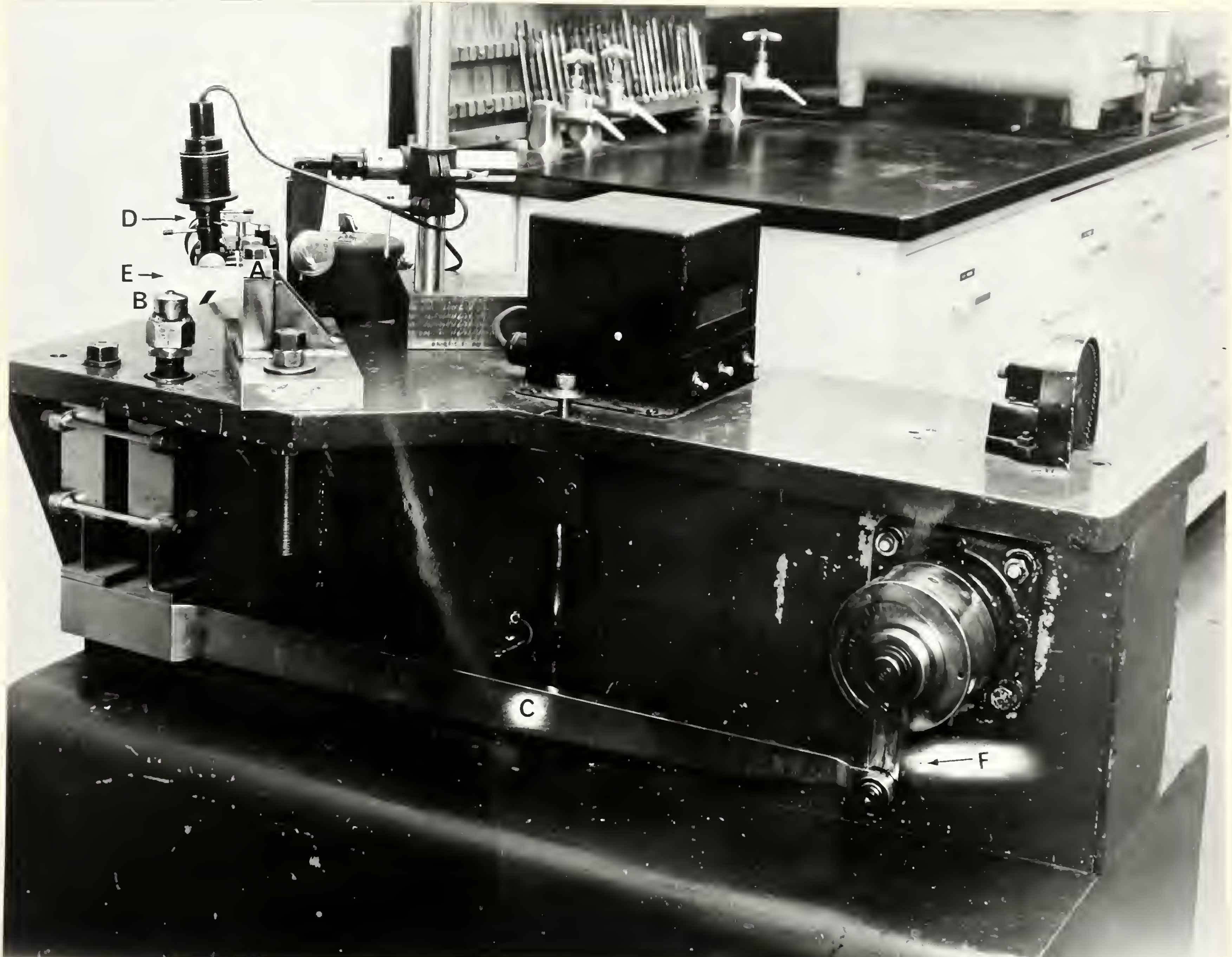
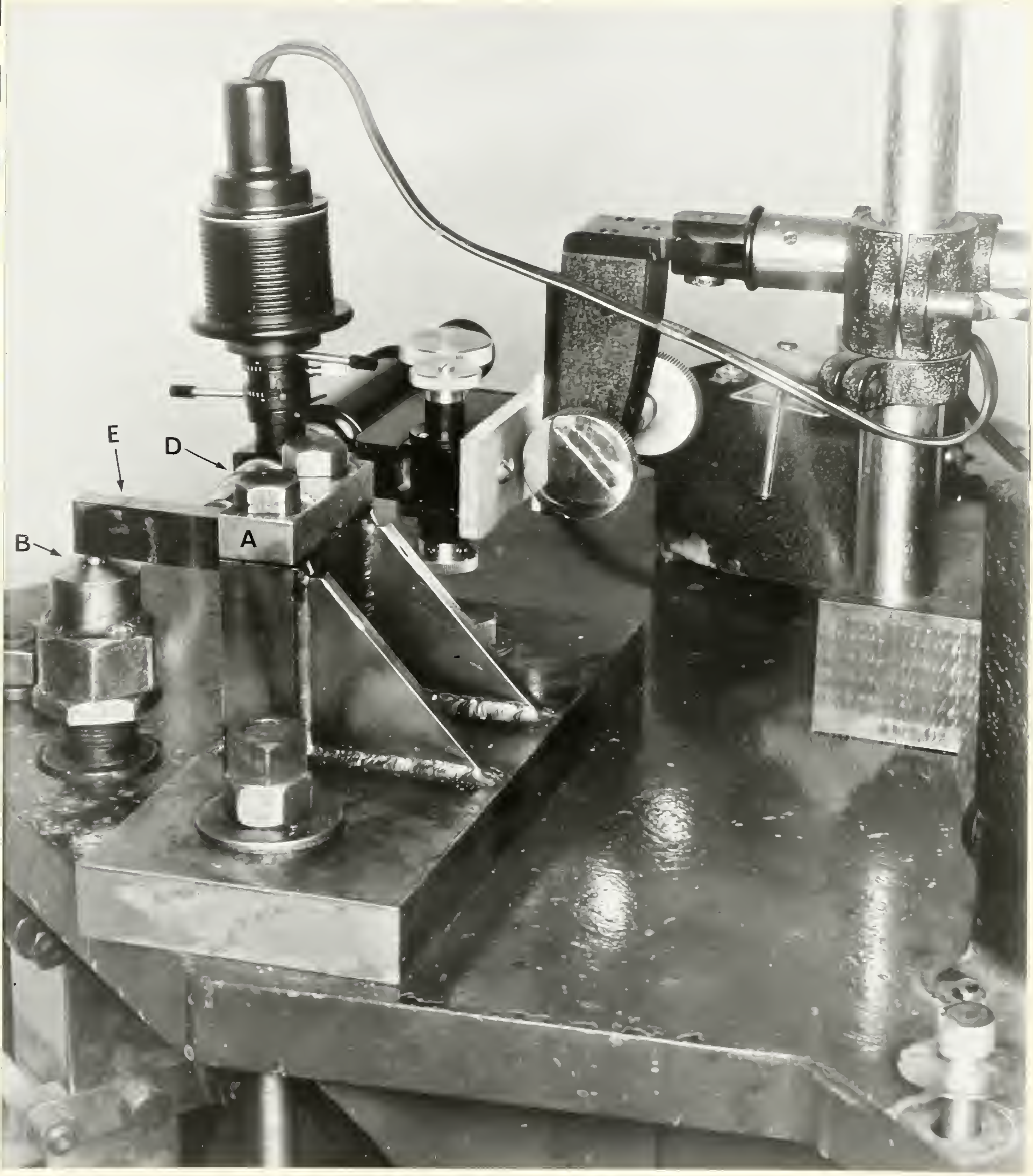

D
E
B
A
C
F

Figure 2. Portion of the area in Figure 1 showing in greater detail, the specimen, arrow E, clamped at A, anvil, arrow B and measuring microscope, arrow D.


E
D
B
A

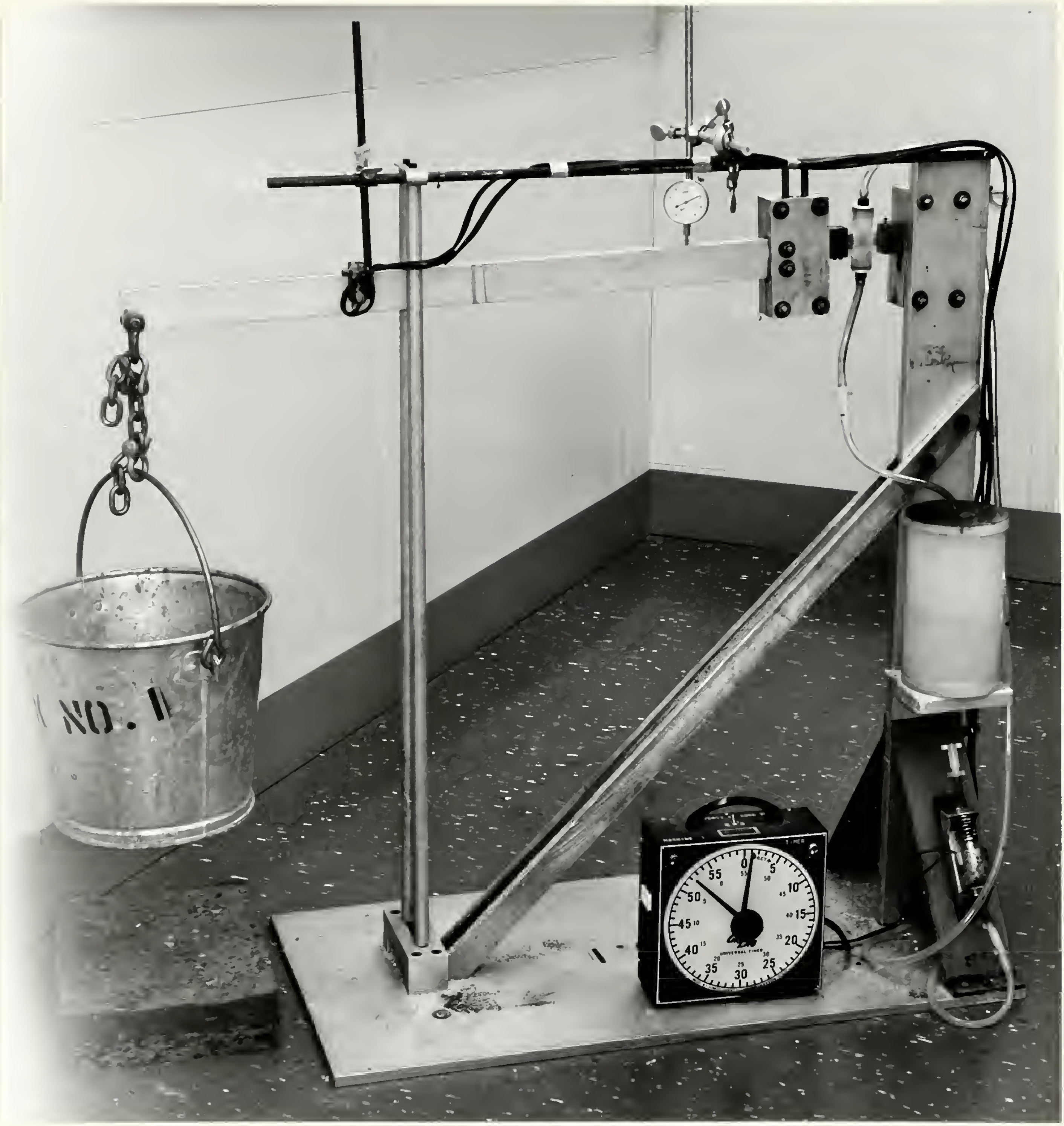


Figure 3. Stress corrosion test apparatus.

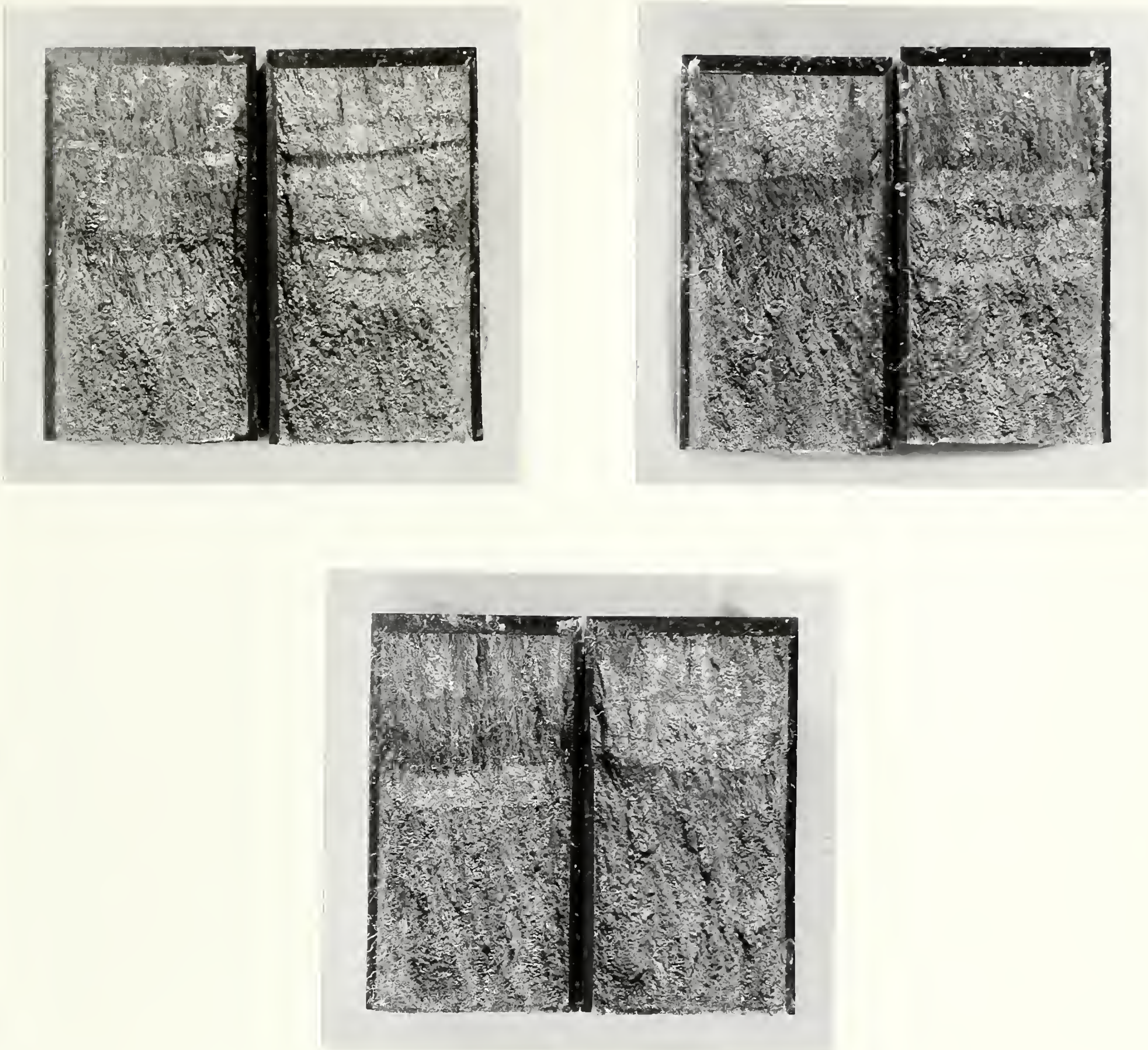


Figure 4. Stress-corrosion test specimens showing typical fractured surfaces. The uniformity of the fatigue cracks (resulting from the pre-cracking process) may be seen in the upper area of each specimen. Each horizontal striation delineates the base of a fatigue crack. The coarser, fibrous areas are those resulting from stress-corrosion and subsequent fast fracture.

DEPARTMENT OF COMMERCE · NATIONAL BUREAU OF STANDARDS